# NR 149.48 Quality control requirements for chemical testing

| Quality Control Measure | Details | Exceptions |
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| (1) General | ➢ Review and update control limits for quality control analyses annually<br>➢ Control limits can be DNR-published, from analytical method, derived in-house or specified by client<br>➢ Fortify quality control samples using a standard from a second source than calibration standards [1]<br>➢ Document identity of preparation batches to ensure compliance with quality control frequencies | [1)] Second source standard is not needed if "additional quality control samples" (blinds)are tested |
| (2) Limits of Detection and Quantitation (LOD and LOQ) | ➢ Determine detection limit annually or when instrument or method is changed<br>➢ Can substitute LOD verification procedure for annual LOD determination<br>➢ Establish procedure to relate LOD to LOQ | Doesn't apply to BOD, solids, O&G-freon, titrimetric tests, or tests where it is not possible to fortify samples |
| (3) Method blank (MB) | ➢ Process blank in same manner as samples<br>➢ Analyze 1 per preparation batch or analytical batch if no preparation step<br>➢ Batch is reanalyzed or qualified if blank is higher of:<br>• Limit of detection, or<br>• 5% of regulatory limit, or<br>• 10% of sample concentration | Doesn't apply to pH, alkalinity, conductivity, or solids |
| (4) Laboratory Control Samples (LCS) | ➢ Process LCS in the same manner as sample<br>➢ Analyze 1 per preparation batch or analytical batch if no preparation step [1]<br>➢ Use a known standard that is from a second source (different from calibration standards) [2]<br>➢ Evaluate LCS results using control limits published by the WDNR, from analytical method or derived in-house<br>➢ Reanalyze or qualify on report associated samples when LCS fails control limits<br>➢ Chlordane, PCBs and toxaphene are treated differently<br>➢ May analyze replicate LCS to determine reproducibility without matrix effects | [1] Matrix spikes can be substituted for laboratory control samples if they are evaluated using LCS acceptance criteria<br>[2] Second source standard is not needed if "additional quality control samples" (blinds)are tested<br>• Doesn't apply to pH, chlorophyll a, color, odor, O&G-Freon, or solids |

Analytical batch as defined in NR 149.03 (7) a set of any number of prepared samples, such as extracts, digestates, or concentrates, or samples requiring no preparatory steps analyzed together as a group in an uninterrupted sequence, and may consist of samples of various quality system matrices.

Preparation batch as defined in NR 149.03 (56) a batch of up to 20 samples, not counting quality control samples, of the same quality system matrix processed in a 24-hour period from the start of the processing of the first sample to the start of the processing of the last sample. In laboratories that do not analyze more than 7 samples for a given test and quality system matrix per week a preparation batch may consist of up to 7 samples, not counting quality control samples, processed over the course of a week.

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| (5) Matrix Spike / Matrix Spike Duplicate (MS/MSD) | ➢ Process matrix spikes and matrix spike duplicates in same manner as samples<br>➢ Analyze 1 per preparation batch per quality systems matrix when required by method or project plan[1]<br>➢ Evaluate MS/MSD results using control limits published by the WDNR, from analytical method or derived in-house<br>➢ Reanalyze or qualify spiked sample result when MS or MSD fails control limits<br>➢ If used in place of LCS, follow LCS corrective action if MS fails | [1] A separate source standard is not needed if "additional quality control samples" are tested (blinds)<br>● Doesn't apply to pH, BOD, CBOD, chlorophyll a, color, odor, O&G-Freon, alkalinity, acidity or solids<br>● Sample duplicate can be substituted for MS duplicate when analyte is likely to be present above LOQ |
| (6) Replicates | ➢ Process replicates in same manner as samples<br>➢ Analyze 1 per preparation batch per quality systems matrix when required by method or project plan<br>➢ Evaluate replicate results using control limits published by the WDNR, from analytical method or derived in-house<br>➢ Reanalyze or qualify spiked sample result when replicate fails control limits | |
| (7) Surrogate spikes | ➢ Add method-specified compounds to all samples and quality control samples at the time of preparation<br>➢ Evaluate surrogate results using control limits published by the WDNR, from analytical method or derived in-house | Applies primarily to chromatography techniques |
| (8) Additional Quality Control Samples (QCS) | ➢ Only required if second source standards are not used to verify initial calibration or to fortify LCS, and MS/MSD<br>➢ Analyze 3 times per year<br>➢ Evaluate QCS results using control limits from the provider | Currently called blinds |
| (9) Selectivity | ➢ Confirmation of organic analytes if not using mass spectrometer as detector<br>➢ Establish procedures for reporting results from dual column and dual detector systems<br>➢ Develop retention time windows acceptance criteria<br>➢ Develop mass spectral tuning acceptance criteria | Applies mainly to chromatography and mass spectral techniques |

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